CHAPTER 1
INTRODUCTION

1.1. Background

Polymers and composite material are an important class of engineering material so modifying and improving properties of them are considerable and interesting for engineers. Also, these types of materials offer excellent mechanical properties, unique flexibility in design and ease of fabrication (Bhagwan & Lawrence, 1990). Composites normally using high strength fibers such as graphite, aramid and glass are commonly used in broad range of applications from aerospace structure to automotive parts and from building materials to sporting goods (Arib, Sapuan, Ahmad, Paridah, & Zaman, 2006). However, the development of natural fiber reinforced composites become an attractive research due to the non degradability, high density and health hazards of composites reinforced with synthetic fibers such as glass, carbon and aramid fibers. Therefore, there has been growing interest in the use of natural cellulosic fibers as the reinforcement for polymeric matrix. Several natural fibers such as sisal (Chow, Xing, & Li, 2007), jute (Ahmed & Vijayarangan, 2007) fibers and so on have been studied as a reinforcement and filler in polymer composites. Green, environmentally friendly, sustainable, renewable, biodegradable, composites from natural fibers are among the most keenly required materials nowadays. Natural fibers reinforced polymers also have exhibited numerous advantages such as high mechanical properties, low weight, low cost, low density, high specific properties (Manfredi, Rodríguez, Wladyka-Przybylak, & Vázquez, 2006), posses better electrical resistance, good thermal and acoustic insulating properties and higher resistance to fracture.
Furthermore, development of polymer made life easier and comfortable and introduce new life to human but recycling polymer which used by human need to recycle. It is true that polymers are recyclable but process of recycling polymers require greater processing such as heat treating, thermal depolymerization and monomer recycling to be recycle due to high molecular weight of their large polymer chain. Polyethylene and polypropylene plastic products such as bags, film and packaging to degrade totally and harmlessly after use, thereby providing the best environmental solution to the plastic pollution problem, at little or no extra cost. So, Eco-polymers provide managerial, technical, marketing and logistical resources and support necessary to ensure efficient and cost-effective.

1.2. Problem statement

Natural fibers can be produced in many types of reinforcement composites, such as continuous and discontinuous unidirectional fibers, random orientation of fibers, etc. By taking the advantages from those types of reinforced composites such as produced good properties and reduced the fabrication cost, they had been used in the development of automotive, packaging and building materials.

There some polymers which are degradable and eco-friendly but those are not good enough for engineering applications. By reinforcing these kinds of polymers, we can achieve more useful material which can use in industrial. In this case there many ways for reinforcing such as woven or randomly mix to get composite of that polymer that can use natural or synthetic fibers. The matrix is often reinforced by stronger and stiffer reinforcing fibers such as carbon, glass and etc. In this project, one type of fiber which natural from
palm and using polyvinyl alcohol (PVA) to get the properties of composite such as hardness, void content, thermal analysis and act of sample and structure of mixture of it.

1.3. Research objectives

Objective of this project include:

1. To prepare polyvinyl alcohol/empty fruit bunch fiber biocomposite by the casting method and fabrication technique with various proportions of fibers
2. To study the effect of empty fruit bunch (EFB) fiber to the mechanical properties, thermal stability, FTIR and morphology of PVA matrix
3. To investigate the effect of EFB fiber to chemical resistance, water absorption and void content of PVA/EFB composite
4. To determine the optimum composition of EFB fiber which provide the optimum reinforcement properties of PVA/EFB composite
CHAPTER 2

LITERATURE REVIEW

2.1. Types of fibers

Fibers are class of hair-like materials that are in discrete elongated pieces, similar to pieces of thread (Natural, 2007). They can be spun into filaments, thread or rope. They can be used as a component of composite materials. Fiber can be classified into two main groups, which are man-made fiber and natural fiber. In general, natural fibers can be subdivided as to their origin such as plants, animals, or minerals; while man-made fibers can be subdivided to synthetic and natural polymers.

The first fibers used by man were natural fibers such as cotton, wool, silk, flax, hemp and sisal. The first man-made fiber was probably glass (Akil et al., 2011). Both natural and synthetic fibers (commonly known as man-made fibers) are now available and always being used as fillers in making a good properties of composites. The major fibers used till now can be classified into the groups given in Figure 2.1.

![Figure 2.1. Classification of fibers (Akil, et al., 2011).](image-url)
Reinforcing fibers in a single-layer composite may be short or long compared to its overall dimensions. The long fibers and short fibers are called continuous fibers and discontinuous fibers, respectively (Agarwal, Broutman, Broutman, & Associates, 1990). The continuous fibers in a single-layer composite may be all aligned in one direction to form a unidirectional composite. The unidirectional composites are very strong in the fiber direction but are generally weak in the direction perpendicular to the fibers. The continuous reinforcement in a single layer may also be provided in a second direction to provide more balanced properties. The bidirectional reinforcement may be provided in a single layer in mutually perpendicular directions as in a woven fabric. The orientation of short or discontinuous fibers cannot be easily controlled in a composite material. In most cases the fibers are assumed to be randomly oriented in the composite (Agarwal, et al., 1990). Alternatively, short fibers, sometimes referred to as chopped fiber may be converted to a lightly bonded perform or mat that can be later impregnated with resin to fabricate single-layer composites. Chopped fibers may also be blended with resins to make a reinforced molding compound. These fibers tend to become oriented parallel to the direction of material flow during a compression or injection molding operation and thus get a preferential orientation. Figure 2.2 represents an accepted classification of single layer alignments.

![Single-layer alignments](image)

Figure 2.2. Single-layer alignments (Agarwal, et al., 1990).
2.1.1 Synthetic fibers

Synthetic fibers are the result of extensive research by scientists to improve upon naturally occurring animal and plant fibers used in making cloth and rope (Synthetic, 2007). A large number of synthetic fibers with a variety of properties have been produced from polymers by various spinning techniques, including melt, dry, wet and emulsion spinning. Before synthetic fibers were developed, artificial (manufactured) fibers were made from cellulose, which comes from plants. At the beginning of the twentieth century, synthetic fibers started supplementing and replacing natural fibers. The first truly synthetic fiber was nylon, followed by polyesters, polyacrylics and polyolefins. Also synthetic elastomeric, glass and aramid fibers became important commercial products (Akil, et al., 2011).

Synthetic fibers are now available, ranging in properties from the high-elongation and low-modulus elastomeric fibers, through the medium-elongation and medium-modulus fibers such as polyamides and polyesters, to the low-elongation, high-modulus carbon, aramid and inorganic fibers (Hannant, 1989). With such a wide variety of synthetic fibers available, the volume of synthetic fibers consumed in worldwide is now greater than that of natural fibers. Most synthetic fibers have relatively smooth surfaces and they are frequently subjected to various mechanical and heat-setting processes to provide crimp (Akil, et al., 2011).

The modern synthetic fiber that was made from older artificial materials and become the most common of all reinforcing fibers for polymer matrix composites is glass fiber (Agarwal, et al., 1990). The principal advantages of glass fibers are low cost and high strength compared with others synthetic fibers. The disadvantages are low modulus and
poor adhesion to polymer matrix resins, particularly in the presence of moisture. The classification of synthetic fibers is shown in Figure 2.3.

![Classification of synthetic fibers](image)

Figure 2.3. Classification of synthetic fibers (Akil, et al., 2011).

### 2.1.2 Natural fibers

Natural fibers are subdivided based on their origins, for example vegetable/plants, animals, or minerals. Figure 2.4 shows the classification of natural fibers. Vegetable or plant fibers include bast or stem fibers, leaf or hard fibers, seed, fruit, wood, cereal straw and other grass fibers(Alexander, Supriya, & Thomas, 2005). Mineral fibers are naturally occurring fiber or slightly modified fiber procured from minerals. Mineral fibers such as asbestos fibers had been used historically for insulating houses.
Natural fibers are now emerging as viable alternatives to glass fibers either alone or combined in composite materials for various applications. The advantages of natural fibers over synthetic or man-made fibers such as glass are their relatively high stiffness, a desirable property in composites, low density, recyclable, biodegradable, renewable raw materials, and their relatively low cost (Joseph, Sreekala, Oommen, Koshy, & Thomas, 2002). Besides, natural fibers are expected to give less health problems for the people producing the composites. Natural fibers do not cause skin irritations and they are not suspected of causing lung cancer (Bos, 2004). The disadvantages are their relatively high moisture sensitivity and their relatively high variability of diameter and length. The abundance of natural fibers combined with the ease of their process ability is an attractive feature, which makes it a covetable substitute for synthetic fibers that are potentially toxic (Liu, Wu, & Zhang, 2009).

Figure 2.4. Classification of natural fibers (Alexander, et al., 2005).

Natural fibers

Vegetable (Cellulose or lignocellulose)  Animal  Mineral fibers
2.2. Natural fiber definition

“Natural” fibers in the strict meaning of the word are produced through agriculture. Natural fibers are a composite material designed by nature. The fibers are basically a rigid, crystalline cellulose microfibril-reinforced amorphous lignin and hemicelluloses matrix. Most plant fibers, except for cotton, are composed of cellulose, hemicelluloses, lignin, waxes and some water-soluble compounds, where cellulose, hemicelluloses and lignin are the major constituents.

From a structural point of view, natural fibers are multicellular in nature, consisting of a number of continuous, mostly cylindrical honeycomb cells which have different sizes, shapes and arrangements for different types of fibers (Dipa & Jogeswari, 2005). These cells are cemented together by an intercellular substance which is isotropic, noncellulosic and ligneous in nature, with a cavity termed the lacuna, whose position and dimensions differ in composition and orientation of cellulosic microfibrils.

There is a central cavity in each cell called the lumen (Dipa & Jogeswari, 2005). The microfibrils in the central walls form a constant angle (microfibrillar or helical angle) for each type of fiber with the fiber axis, so that the crystallites are arranged in a spiral form, the pitch of which varies from one fiber to another. Thus, each fiber is a ‘natural composite’ by itself, wherein crystallites (mostly cellulosic) remain rooted in a matrix in a given orientation. Hence the properties of the single fibers depend on the crystallite content, their sizes, shape, orientation, length/diameter (L/D) ratio of cells, thickness of cell walls, and finally, their defects such as lumen and lacuna (Dipa & Jogeswari, 2005).

Dipa and Jogeswari (2005) summarized that the most important factor controlling the different types of natural fibers is their species because the properties of fibers are different between different species. In addition, the properties of fibers within a species vary
depending on area of growth, climate and age of the plant. Lastly, the properties of natural fibers vary greatly depending on their processing method used to break down to the fiber level.

### 2.3 Classification of vegetable or plant fibers

Vegetable or plant-based natural fibers are lignocellusic, consisting of cellulose microfibrils in an amorphous matrix of lignin and hemicellulose. They consist of several hollow fibrils which run all along their length (Sanadi, Prasad, & Rohatgi, 1986). Each fibril exhibits a complex layered structure, with a thin primary wall encircling a thicker secondary layer, and is similar to the structure of a single wood-pulp fiber. Nishino (2004) found that the primary (outer) cell wall was usually very thin (< 1 μm). The secondary layer is made up of the three distinct layers, the middle one (also known as S2 layer) being by far the thickest and the most important in determining mechanical properties. Sanadi (1989) point out that in this layer, parallel cellulose microfibrils are wound helically around the fibrils which the angle between the fiber axis and the microfibrils is termed the microfibril angle. Natural fibers are themselves cellulose-fiber-reinforced materials in which the microfibril angle and cellulose content determine the mechanical behavior of the fiber.

For fiber geometry, man-made fibers emerging from a spinneret are cylindrical, with approximately constant diameter and specific area. These defects are apparent as ‘knees’ at the fiber surface and constitute points where the fiber may rupture more easily. In addition, an important parameter is the aspect ratio (length/diameter), which has an influence on the mechanical properties of the composite. This aspect ratio is highly modified by attrition during processing such as extrusion or injection.
According to Seena (2005), vegetable fibers can be generally classified as bast, leaf, seed-hair, cereal straw or grass fibers, depending on their origin. Bos (2004) had illustrated the classification of vegetable fibers as shown in Fig. 2.5. In the plant, the bast and leaf fibers lend mechanical support to the plant’s stem and leaf, respectively. Bast consists of a wood core surrounded by stem. Within the stem, there are a number of fiber bundles, each containing individual fiber cells or filaments. Bast fibers are usually grown in warm climates such as hemp, flax, jute, kenaf and ramie (Joseph, et al., 2002). While for leaf fibers such as sisal, abaca, banana and henequen, they are coarser than bast fibers. In contrast, seed-hair fibers, such as cotton, coir and milkweed, are attached to the plant’s seeds and aid in wind dispersal (Seena, Maya, & Sabu, 2005). Joseph et al. (2002) reported that cotton is the most common seed fiber and it is used for textile all over the world. Historically, humans have used all these natural fibers for textiles because of their wide availability, long fiber geometries, chemical stability, and favorable mechanical properties. The coarse texture and high mechanical properties of many bast and leaf fibers have made them common cordage fibers for rope, twine, and string (Seena, et al., 2005).

Figure 2.5. Classification of vegetable or plant fibers (Bos, 2004).
2.3.1 Bast and core fibers

Bast fibers exist in the inner bark or phloem of many dicotyledonous plants to provide structural rigidity to the stems. These fibers occur in bundles which run parallel to the stems between nodes. The fiber strands are composed of many smaller cells termed ultimate fibers. Just inside the phloem is a wood-like core material consisting of short and fine fibers (Seena, et al., 2005). According to Alexander et al. (2005), the shape and size of the stem of various bast fiber crops are different but they all contain varying amount of fiber cells in the phloem. Long individual fiber or long fibers bundles can be obtained from many bast fiber crops at relatively low cost.

However, bast fibers have a non-homogeneous cell structure than do the much shorter wood fibers which are uniform, readily available and inexpensive. Bast fibers have been used for generations to produce textiles and are removed from the phloem by a controlled decay and separation process called retting. In general, the core material is unused after the outer bast fibers are removed (Seena, et al., 2005). Bast fiber crops have rigid herbaceous stalks containing nodes at regular intervals that are fluted or channeled. From the inside to outside the stalks have a hollow core, except at joints, followed by the pith and finally the protective layer epidermis (Alexander, et al., 2005).

Characteristic geometries and dimensions for bast and core fibers are compiled in Table 2.1. In general, bast fibers strands are large with the average length of 25 to 120 cm and widths less than 1 mm. The ultimate fibers comprising these strands range in size (Seena, et al., 2005). For jute and kenaf, the ultimate fibers have a similar size to many coniferous wood species. However, for flax and hemp, they have extremely long ultimate fibers that are only minimally larger in diameter to wood. These fibers typically have aspect ratios (length/diameter) on the order of 1000.
2.3.2 Leaf fibers

In many monocotyledons, long strand leaf fibers are embedded in parenchymatous tissue to provide mechanical support for the long and broad leaves. As in the bast, these composite fiber strands are composed of many smaller ultimate fibers (Seena, et al., 2005). In general, leaf fibers are more coarse than bast fibers and are commonly used as cordage, mats, rugs, and carpet backings rather than clothing fabrics. Common examples of these materials are Manila hemp (abaca) and sisal, both of which are used for twines and ropes worldwide. In addition, abaca and sisal have been both used historically for paper fibers (Seena, et al., 2005). According to Alexander et al. (2005), abaca and sisal are hard fibers obtained from the leaves of their plant and they are considered to be the strongest of all plant fibers.

2.3.3 Seed-hair fibers

Unlike the bast and leaf fibers, seed-hair fibers are single celled. These fibers are attached to the seeds of certain plants for aid in wind-dispersal (Seena, et al., 2005). One exception to this is coir, a fiber produced from the husk of coconuts. Like bast fibers, coir is produced by separation technique after husks undergo retting. Coir can produce long fiber strands, depending on the production process. The ultimate coir fibers are quite small which are less than 1 mm long and 6 mm in diameter.

2.3.4 Cereal straws

Straw is produced throughout the world in enormous quantities as a by-product of cereal cultivation. Half the straw is simply burned on the fields just to get rid of it or else it is
buried in greater quantities than what is needed to replenish organic matter in the soil (Alexander, et al., 2005). Historically, straws from wheat, rye, and rice were widely used as a pulp source for paper-making. Whereas this practice became extinct in North America and much of Europe by 1960, it is still practiced in southern and eastern Europe as well as many Asian, Mid-Eastern, and Southern American countries. Although corn stalks are widely available and have been studied as a pulp source, their commercial use has been limited (Foyle T, 2007; Seena, et al., 2005).

2.3.5 Grass fibers

Many other grasses have been and are considered as a fiber source. Because of limited availability or processing difficulties, most of these fibers (except sugarcane bagasse) have never become widely used; however, they are often common in certain localities. These fibers include sugarcane bagasse, bamboo, esparto, and sabai grass.

In these grass fibers, sugarcane bagasse is the most common and widely studied among the researchers. Sugarcane is grown as a source of sucrose in many tropical and sub-tropical countries on many continents. Bagasse is the residue remaining after the sugar has been extracted (Seena, et al., 2005). Currently, this material is used for paper in India and Mexico and in boilers to generate process steam for the sugar production. Paper and material fiber provide the more economical use for this material (Seena, et al., 2005). The bagasse consists of pith and rind materials. The rind consists of 50% of the dry bagasse weight and contains most of the usable fiber. In general, these fibers are similar to hardwood fibers average 2.8 mm in length and 34 mm in diameter, producing and aspect ratio of 82. Two grasses, esparto and sabai, are widely available in North Africa and India, respectively. Both grasses are locally common as a paper fiber, with sabai at one time
comprising 20% of the pulped material in India. Compared to softwood fibers, these grass fibers have small diameters (9 mm) and average lengths (1-2 mm), producing fibers with large aspect ratios between 100 and 200 (Seena, et al., 2005).

2.4. Biocomposites

A biocomposite is a material formed by a matrix (resin) and a reinforcement of natural fibers (usually derived from plants or cellulose). With wide-ranging uses from environment-friendly biodegradable composites to biomedical composites for drug/gene delivery, tissue engineering applications and cosmetic orthodontics. They often mimic the structures of the living materials involved in the process in addition to the strengthening properties of the matrix that was used but still providing biocompatibility, e.g. in creating scaffolds in bone tissue engineering. Those markets are significantly rising, mainly because of the increase in oil price, and recycling and environment necessities.

Biocomposites are characterized by the fact if the petrochemical resin is replaced by a vegetable or animal resin or the bolsters such as fiberglass, carbon fiber or talc which replaced by natural fibers like wood fibers, hemp, flax, sisal, jute and etc (biocomposite, 2009).

Cañigueral et al (Cañigueral et al., 2009) studied the intrinsic mechanical properties of the reinforcement were measured and the behavior of the obtained matrix which is a starch-based biopolymer was reinforced with flax strands. The biocomposite was obtained by injection molding processing and mechanical properties evaluated as well that show a significant increase of the strength and stiffness of matrix with the percentage of non-modified flax strands. Furthermore, the intrinsic flax fiber tensile strength decreased with
the fiber length due to the presence of critical defects like pits and nodes. The measured intrinsic tensile properties of flax fibers are close to those found in the bibliography.

Wen-Long Dai (Dai, 2003) has used environmental material which is composite of rice-hush (RH) and polyvinyl alcohol (PVA). In an experiment BP-type PVA pellets and water (1:4 mixture ratio in weight) are blended for 170 min under the control of blade speed and then cooled down at room temperature and used to blend RH powders. It is observed that the viscosity value increases sharply as a temperature reaches 60-80 °C. Based on mixing and forming RHP mixture water content plays a significant role for decreasing glass-transition temperature and mold forming can be enhance under suitable control of water and for obtain better bonding and forming the limitation of size different for the mixing of RHP powders is noticeable.

Chun-Chen Yang et al (Yang & Wu, 2009) studied a microporous polyvinyl alcohol (PVA) and polyvinyl chloride (PVC) composite which prepared by solution casting method and a preferential dissolution method. The proper weight ratio of PVA:PVC(=1:0.025-0.1) polymers were dissolved in hot water that was stirred until obtain homogenous mixture appearance at 85-90°C for 30-60 min. The glass plate with viscous PVA/PVC composite polymer membrane was weighed again and then the excess water was allowed to evaporate slowly at a constant temperature of 50 °C at relative humidity of 30RH%. After water evaporation, the glass plate with the composite polymer membrane was weighed again. The composition of PVA/PVC composite polymer electrolyte membrane was determined from the mass balance. After prepared composite for required size, characteristic of it studied X-ray diffraction (XRD), scanning electron microscopy (SEM), thermal gravimetric analysis (TGA) and etc. Result of experiment showing excellet thermal property, dimensional stability and ionic conductivity and finally concluded that this composite is a good candidate for $MnO_2$ capacitors.
Based on reference (Xuefeng & Shaoxian, 2011) Xuefeng Li studied the tribological properties of polyvinyl alcohol-carbon nanotubes (PVA-CNTs) composites. A preparation of PVA-CNTs is the mixture of 10 ml of vinyl acetate, 50 ml distilled water, 100 mg sodium dodecylsulfate, 0.1 ml emulsifier OP10 and 50 mg potassium persulfate (initiator) was charged into a 250 ml reaction flask and it stirred for 1.5 hour at 65°C. After adding CNTs and stirred for 2.5 hours, polymeric product dried under vacuum at 50°C in a constant weight. Afterwards, four ball test provided and results shows that as a lubricant additive in water-based fluid, PVA-CNTs can improve the wear resistance, load-carrying capacity and anti-wear ability of base stock.

Maria S.Peresin et al (S.Peresin) has studied fiber nanocomposite of PVA reinforce with cellulose nanocrystals (CN) which prepared by electrospinning technique. Results illustrate the enhancement of the thermo-mechanical properties of CNs inside the PVA nanofiber matrixes and also stabilized the system at high moisture contents.

H. Ku et al (Ku, Wang, Pattarachaiyakoop, & Trada, 2011) mentioned that in last decades natural fibers are alternative reinforcement in polymer composites due to their advantages over conventional glass and carbon fibers. In general, the Young’s modulus of the composite materials increase with an increase in fiber content, reaching a maximum value at 50% hemp fiber loading and then decreasing slightly at 70% hemp fiber content. The Young’s modulus was almost two and a half times higher at 50% hemp fiber loading than at 0% fiber content. It shows many works which done by researcher and compared together for showing properties of natural fibers.

Sathishkumar et al (Sathishkumar, Navaneethakrishnan, & Shankar, 2012) by considering the cost of weight effectiveness of natural fibers showing plenty advantages of them over synthetic fibers. He reinforced thermoset isophthallic polyester resin by various volume of snake grass fiber. The tensile strength of the fiber was obtained by the ratio of average load
to the average area for the twenty-five identical samples. Study illustrates that when the
fiber length is increased beyond a limit, then the tensile modulus value gets decreased. This
shows that the fiber length has influential effect in setting the tensile modulus value of any
natural fiber composites. The maximum flexural strength and modulus of the chopped fiber
isophthalic polyester composite is achieved at 25% $V_f$ for 120 mm and 150 mm fiber
lengths. But the values have significant improvement in 150 mm. The SEM micrograph of
tensile and flexural tested specimens predicts the fiber failure, matrix crack and fibers pull
out during the loading condition at 10% $V_f$ and 25% $V_f$ of the composite. At higher strength,
the volume fraction composite has less fiber pull out due to the more accumulation of fiber
being wetted in matrix and also it transfers higher load. Overall, it can be concluded that the
25% volume fraction of the snake grass fibers composite have the maximum mechanical
properties.

Maha M. Ibrahim et al (Ibrahim, Dufresne, El-Zawawy, & Agblevor, 2010) lignocellulosic fillers extracted from banana plant waste were prepared from either an
alkaline pulping treatment or steam explosion. They were used to reinforce a polyethylene
matrix. With maleic anhydride, the particle size was reduced for the steam exploded banana
fiber compared to the alkaline-pulped banana fiber. The addition of 20% malleated fiber
results in a better adhesion with the polymer matrix, and thus better tensile strength, while
for further fiber concentration it seems to lose its flexibility and lead to poor adhesion with
the polymer matrix.

Yousif et al (Yousif, Lau, & McWilliam, 2010) studied and tested betel nut fiber
reinforced polyester composite (BFRP) which was fabricated using hand lay-up technique.
Tensile and pull out test and tribological tests was included and analysis and micrograph of
specimen provided as well. Results showing that under wet condition, the tensile strength
of the fiber are reduced by about 17% compared to the dry. However; the wet fiber has higher strain than the dry by about 26%. Under dry condition, the interfacial adhesion of the fiber is high compared to the wet, i.e. there was no pull out.

Sinto Jacob et al (Jacob, Suma, Mendez, & George, 2010) study effect of nanosilica on polypropylene which modified by nylon fiber. Composite prepared and mixed by using different weight percentage of nanosilica on polypropylene-nylon fiber in a Thermo Haake Rheocord. Afterwards, comparison and discussion of result provided as well which showing composite of 1 wt.% modified nanosilica to PP–10 wt.% nylon fiber preferable compare to others.

Vail et al (Vail, Krick, Marchman, & Sawyer, 2011) used Polytrafluoroethylene (PTFE) fiber to reinforce polyetheretherketone (PEEK) and studied tribological properties of composite. The sample was evaluated by linear reciprocating tribometer and microscope image of tested sample provided. Result showing that the inclusion of PTFE in PEEK dramatically decreases both the friction coefficient and the wear rate of the base material. Also, expanded PTFE fibers of 5mm or longer have sufficient surface area in contact with the PEEK to reinforce the matrix and resist being pulled out.

Yamamota et al (Yamamoto & Hashimoto, 2004) studied and tested the friction and wear characteristic of fiber reinforced PEEK and Polyphenylenesulphide(PPS) in water.

2.5. Biopolymer

Biopolymers are polymers produced by living organisms, and most biopolymer applications are in the field of medical engineering, such as drug delivery systems, wound healing, and surgical implantations. With the aim of environmentally friendly development, biopolymers such as welan gum and curdlan have been used as bio-admixtures in concrete
or dry-mix mortar as water-retention agents or superplasticizers according to their pseudo plasticity properties. In the fields of soil science, geotechnical engineering, and geoenvironmental engineering, biopolymers have been applied as soil stabilizers in order to control or reduce soil erosion and are used for soil drilling mud and temporary excavation supports. Aligning with this trend, theoretical and experimental verifications of the interactions between various types of biopolymers and soil media are required in the geotechnical and geo-environmental fields (Chang & Cho, 2012).

2.5.1. Polyvinyl alcohol (PVA)

Polyvinyl alcohol (PVA) is a water-soluble which it has excellent film forming, emulsifying and adhesive properties. It is also resistant to oil, grease and solvents. Furthermore, the PVA polymer used is biodegradable, nonhazardous, and environmentally benign (Anis, Banthia, & Bandyopadhyay, 2008).

PVA it uses for thickener, modifier, in polyvinyl acetate glues, textile sizing agent, Paper coatings, release liner, Carbon dioxide barrier in polyethylene terephthalate (PET) bottles, as a mold release because materials such as epoxy do not stick to it and etc. Recently, nano composite membrane of PVA using in direct methanol fuel cell (DMFC) which the membrane shows good thermal properties (Maiti, Kakati, Lee, Jee, & Yoon, 2011).

2.5.2. Environmental Impacts of Biopolymers

Biopolymers can be sustainable, carbon neutral and are always renewable, because they are made from plant materials which can be grown indefinitely. These plant materials come from agricultural non food crops. Therefore, the use of biopolymers would create a
sustainable industry. In contrast, the feedstocks for polymers derived from petrochemicals will eventually deplete. In addition, biopolymers have the potential to cut carbon emissions and reduce CO2 quantities in the atmosphere: this is because the CO2 released when they degrade can be reabsorbed by crops grown to replace them: this makes them close to carbon neutral (biopolymer, 2012).

Biopolymers are biodegradable, and some are also compostable. Some biopolymers are biodegradable: they are broken down into CO2 and water by microorganisms. Some of these biodegradable biopolymers are compostable: they can be put into an industrial composting process and will break down by 90% within six months. Biopolymers that do this can be marked with a 'compostable' symbol, under European Standard EN 13432 (2000). Packaging marked with this symbol can be put into industrial composting processes and will break down within six months or less. An example of a compostable polymer is PLA film under 20μm thick: films which are thicker than that do not qualify as compostable, even though they are biodegradable. In Europe there is a home composting standard and associated logo that enables consumers to identify and dispose of packaging in their compost heap (NNFCC, 2010).

2.6. Application of biocomposite and biopolymers

2.6.1. General and engineering application of biocomposites

Various biocomposites depend on matrix can be very cost effective material compare to other composites in general application such as storage devices (post-boxes, grain storage silo, bio-gas containers) and furniture like chair, table and etc and electric devices (electric appliance, wires cover etc).
The latest research on biocomposites had been develop wide range of engineering application and industrial. Several biocomposites products are being used in a large variety of engineering purposes:

i. As environmental concern solutions, automotive industry has shown increased interests in seeking wide application of natural or bio-based materials in recent years. Table 2.1 shows automotive industry with the different uses of fiber sources.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Fiber source</th>
<th>Parts</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volvo</td>
<td>Hemp, jute</td>
<td>Dashboards, ceiling, seat filling, cargo floor tray</td>
<td>Current-future</td>
</tr>
<tr>
<td>Toyota</td>
<td>Kenaf</td>
<td>Door trim</td>
<td>Current</td>
</tr>
<tr>
<td>Ford</td>
<td>Kenaf</td>
<td>Door trim, trunk liner</td>
<td>Current</td>
</tr>
<tr>
<td>Daimler Chrysler</td>
<td>Flax, hemp, sisal, coconut</td>
<td>Door cladding, seatback lining, package shelves, seat bottom etc (&gt;50 parts)</td>
<td>Current</td>
</tr>
<tr>
<td>Mercedes</td>
<td>Flax, hemp, sisal</td>
<td>50 components-42.7 kg vs 24.6 kg in earlier model (+73%)</td>
<td>Current</td>
</tr>
</tbody>
</table>

ii. Fiber cement composites largely apply in housing construction for exterior and interior building such as floors, wall fencing, roofing cladding and etc (Jamaludin Mohamad Yatim, 2011). Also, other application can use for repair and rehabilitation of structural element which can be apply at bridge, column, slab and beam column joint and so on.
2.6.2. Application of biopolymers

Applications of biodegradable polymers and biopolymers can be found in the medical field and can be roughly divided into three categories: drug delivery systems, wound closure and healing products, and surgical implant devices. Drug delivery inside the human body can be quite easily controlled with the use of biodegradable capsules. In wound healing, resorbable non-wovens for the replacement of human tissue, as well as simple sutures, staples, clips or meshes are available. Related to these applications, also the use as bioresorbable scaffolds for tissue engineering is worth mentioning (Penning, Dijkstra, & Pennings, 1993).

Other applications are numerous. Many of the biodegradable polymers have good film forming properties, making them suitable for applications in high performance applications as well as in traditional commodity uses. Some applications include food containers, soil retention sheeting, agriculture film, waste bags and the use as packaging material in general. When used as non-wovens, these biopolymers can also be used in agriculture, filtration, hygiene and protective clothing (Van de Velde & Kickens, 2002).
CHAPTER 3
METHODOLOGY

3.1. Materials

Oil palm empty fruit bunch (OPEFB) fiber in powder form was used and purchased from the Malaysian palm Oil Board in Bangi, Selangor, Malaysia. Polyvinyl alcohol (PVA) purchased from Kuraray Asia Pacific Pte. Ltd in Singapore which material in liquid form contain 94% PVA and remain water and methyl acetate and methanol.

3.2. Sample preparation

Preparation of fiber reinforced PVA was prepared by a solution casting method and preferential dissolution process. The appropriate weight ratios of PVA liquid and palm fiber added to the liquid form by 2 and 5 percentage of weight of PVA. So, by weighting appropriate PVA and calculate of 2 and 5 percentage of it, can get exact amount of fiber weight to add on.

The addition of fibers in different amount (percentages) and in the glass vessel was slowly and well controlled to get uniform distribution of PVA/EFB matrix. Afterwards, the resulting mixture solution was poured out into a glass plate and for evaporating excess water it kept in oven in constant temperature of 50 ° C for 48 hours. The polymer has high sticky properties that using of mold release agent is necessary. After water evaporation, dry PVA and composites of it poured into hot mount press machine (Specimen mount press 20-1310, BUEHLER Ltd ). After poured dried sample into machine, it heated for 5min till
80°C C which it was controlling by thermometer and after it pressed and kept it to cool down to eject sample. The process repeated for two others composite in same way to get three samples in circle shape.

Figure 3.1. Samples after process a) pure PVA, b) PVA/EFB-2%, c) PVA/EFB-5%

3.3. Testing and characterization

3.3.1. Morphology of fiber and sample

The palm fibers and the composites were observed using FESEM on the surfaces of two sample composite to see how fibers distributed in composite. Samples with uniform distributed fiber in composite showing more accurate results for other tests.
3.3.2. Chemical resistance properties

The chemical resistance of PVA and its composites (PVA/EFB-2% and PVA/EFB-5%) were studied using ASTM D 543-87 method. In test effect of some solvents i.e. toluene, benzene, the effect of some alkalies i.e., NaOH, Na2CO3, NH4OH and the effect of hydrochloric acid were studied on the pure and matrix composites samples. In each case three pre weighed samples were dipped in the respective chemical reagents for 24 h. They were then removed and immediately washed in distilled water and dried by pressing them on both sides with a filter paper at room temperature. The samples were then weighed and the percentage weight loss/gain was determined. By using the following equation the percentage of weight loss/gain was determined.

\[
\text{Weight loss (\%)} = \frac{Final \ weight - Original \ weight}{Final \ weight}
\]  

(3.1)

3.3.3. Water absorption

For determination of water absorption in composites, ASTM D 570 [25] method was used. Test samples were dried in the oven for 24 h at 50°C, cooled in a desiccators and immediately weighed for the conditioning steps. Water absorption tests were conducted for 1 h immersion and reconditioned with same conditioning step. The sample tests were reconditioned due to the presence of water-soluble matter in the composites.
3.3.4. FTIR measurements

FTIR measurements were carried out using a single beam Fourier transform infrared spectrometer (FTIR-430, Jasco, Japan). The FTIR spectra of the samples were obtained in the spectral range from 3545 to 595 cm$^{-1}$. All samples were prepared using a thin film from each of samples and pure fiber in powder. The FTIR spectra were processed using Jasco Spectra manager software. The peak areas and the absorbance ratios were obtained accurately by using this software.

3.3.5. Void content

For determination of voids in composites, ASTM 2734-94 (2734-94, 1997) method was used. The void content was determined from the theoretical and experimental density of the composite through equation (3.2).

$$\text{Void content} = \frac{\rho_{\text{theoretical}} - \rho_{\text{experimental}}}{\rho_{\text{experimental}}}$$  \hspace{1cm} (3.2)

Where

$$\rho_{\text{theoretical}} = \frac{1}{\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}}$$  \hspace{1cm} (3.3)

Where $w_f$ is the fiber weight fraction, $w_m$ the matrix weight fraction, $\rho_f$ the density of fiber, and $\rho_m$ is the matrix density.

In this part of test, the sample by 4% fiber was made to compare with two others sample for more accuracy.
3.3.6. Hardness and yield strength

Hardness is simple, but useful test to characterize the mechanical properties of material. Hardness of material express that how it resistant to various kinds of permanent shape change when a force is applied and generally defined as resistance to penetration by an indenter, though resistance to scratch is also used. It is long time that Vickers hardness test is using for metallic materials and ceramics but only recently has it been widely employed for characterizing polymers (Suwanprateeb, 1998).

Polymers are viscoelastic materials so their behavior is time-dependent. In this study, test was performed by two different loads in three different time. The hardness decreased as the time elapsed. This is analogous to creep behavior when the materials deform under constant load.

The samples were indented at 19 °C and 48% RH by using hardness tester (Shimadzu micro hardness tester HMV-2) with Vickers diamond pyramidal indenter having square base and pyramidal angle of 136°. There were measured manually using an electronic ruler on the projected screen where an indentation image was displayed through objective lenses. Vickers hardness number was calculated using equation (3.4):

\[ H_{v,t} = 1.854 \frac{r}{d^2} \]  \hspace{1cm} (3.4)

In next step due to difficulty of tensile test for achieve yield strength we going to predict tensile strength from hardness Vickers (HV). Designers and users of cold formed products need to know usually the new increased yield strength of it as accurate as possible. However, the yield strength distribution is inhomogeneous in the product and the products are in general not appropriate for standard material testing experiments like simple tension. Therefore, usually either simplified analytic computations or finite element models are used.
to predict the new yield stress distribution. Alternatively, the local new yield stress is determined by means of hardness measurements, which are converted to the respective flow stress at the point of measurement.

According to (Tekkaya & Lange, 2000) which is improved relationship between Vickers hardness and yield strength can be given as.

\[ Y_{rep} = \frac{9.81 \times HV}{2.475} \]  \hspace{1cm} (3.5)

Which compute the representative flow stress (in MPa).

3.3.7. Thermal analysis

Polymers represent another large area in which thermal analysis finds strong applications. Thermoplastic polymers are commonly found in everyday packaging and household items, but for the analysis of the raw materials, effects of the many additive used and fine-tuning of the molding or extrusion processing used can be achieved by using Differential scanning calorimetry (DSC) and Thermo gravimetric analysis (TGA).

The basic principle of DSC technique is that when the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to it than the reference to maintain both at the same temperature. DSC is used widely for examining polymeric materials to determine their thermal transitions.

The DSC test was performed using a differential scanning calorimeter (Mettler Toledo). Overall, 12–13mg of the samples were loaded in a differential scanning
calorimetry (DSC) pan, and the pan was sealed by applying pressure. The samples was heated from 30°C to 250°C. Experiments were repeated twice for reproducibility.

TGA can be used to evaluate the thermal stability of a material. In a desired temperature range, if a species is thermally stable, there will be no observed mass change. Negligible mass loss corresponds to little or no slope in the TGA trace. TGA also gives the upper use temperature of a material. Beyond this temperature the material will begin to degrade. TGA has a wide variety of applications, including analysis of ceramics and thermally stable polymers. Ceramics usually melt before they decompose as they are thermally stable over a large temperature range, thus TGA is mainly used to investigate the thermal stability of polymers.

The TGA test was performed using a differential scanning calorimeter (Mettler Toledo). The samples were loaded in a Thermo gravimetric analysis (TGA) pan. The samples were heated from 35°C to 1000°C in a constant heat rate.
4.1. Morphology study

The palm oil fibers and the composites were observed using Field Emission Scanning Electron Microscopy (FESEM). The samples which prepared in same size cut by cutter to observe section of sample and surface which is shows in figure 4.1. The pictures showing the surface of samples and a section part in a sample.
Figure 4.1. FESEM micrographs: In two samples (a) surface of 2% fiber (b) surface of 5% 
(c) random fiber size (d) section of sample 5%

As it taken from FESEM pictures sizes of fibers is in different width and length (in fig4.1.c 
randomly size of fiber is 771.4 μm × 45.99 μm) and distribution of fibers are random. 
Also, it is visible and clear that we can see too much fiber inside 5% compare to 2% fiber. 
In section part of sample fig 4.1(d) can see as it cut fibers lost their original shape and 
surface roughness of samples increase due to cut so, some surface treatments required as it 
cut to reduce the roughness of composite samples.

4.2. Chemical resistance test

Chemical resistance tests are used to find the ability of a composite to withstand exposure 
to acids, alkalis, solvents and other chemicals. The chemical resistance tests of composites 
were performed in order to find out whether these composites can be used for making
articles that are resistant to chemicals. The weight loss/gain for the PVA, PVA/EFB-2% and PVA/EFB-5% composites with different chemicals were shown in Table 4.1. The table shows that in the most cases except HCL weight gain is observed. This clearly indicates that the composites did not lose weight, and therefore it does not seem as if any erosion occurred.

Table 4.1. Chemical resistance properties of PVA and its composites.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>PVA</th>
<th>PVA/EFB-2%</th>
<th>PVA/EFB-5%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>1.729</td>
<td>1.9317</td>
<td>1.7481</td>
</tr>
<tr>
<td>Toluene</td>
<td>2.3124</td>
<td>5.3289</td>
<td>7.7634</td>
</tr>
<tr>
<td>H₂O</td>
<td>2.7887</td>
<td>6.1034</td>
<td>6.2301</td>
</tr>
<tr>
<td>HCL</td>
<td>NIL</td>
<td>NIL</td>
<td>NIL</td>
</tr>
<tr>
<td>NaOH (10%)</td>
<td>6.521</td>
<td>11.8994</td>
<td>10.89324</td>
</tr>
<tr>
<td>Na₂CO₃ (20%)</td>
<td>9.5348</td>
<td>11.4595</td>
<td>12.34567</td>
</tr>
<tr>
<td>NH₄OH (10%)</td>
<td>2.749</td>
<td>3.8029</td>
<td>3.542</td>
</tr>
</tbody>
</table>

The weight increase of the samples was larger for composites this was to be expected as a result of the hydrophilicity of the fiber (Guduri, Rajulu, & Luyt, 2007). The PVA and its composites are completely solvate in HCL during 24 hours which means the fibers not effected on resistance of material. The weight gain (%) was roughly more when fiber content of composite and this is due to increase of the hydrophilicity of the system because of fibers.

Totally it was observed that matrix showed better chemical resistant to all chemicals except resistance to HCL which it solvate in it.
4.3. Water absorption test

Water absorption test is important to determine the water absorptivity of the new material and composites due to application them in industry or etc. Figure 4.2 shows the water absorption of the composites for pure PVA samples and its composites by 2 & 5% of fiber contents. From the Figure, water absorption value of the composite increased with higher content of fibers due to the properties hydrophilicity of fibers but the change of water absorption in PVA/EFB-5% is higher than PVA and PVA/EFB-2% samples. It means as it shown PVA/EFB-2% and PVA samples showing almost the same properties of water absorption. It can say that, the content of 2% fiber is not effect too much for water absorption. By referring to the figure the percentage of change in absorption between matrix of 5% and others is about 11% but the change of PVA to PVA/EFB-2% is less than 1%.

![Figure 4.2. Effect of immersion time to the water absorption due to fiber content.](image-url)
**FTIR measurements**

Figure 4.3 shows the FTIR spectra of PVA and its composites with 2&5% of fiber content and pure fiber in powder. It is clear in figure that in all of the absorption peaks both of the PVA composites showing less absorbance due to fiber content. The different of absorption after the frequencies of about 1680 cm$^{-1}$ become more clear and between the frequencies 1125 and 980 the peak drop dramatically which it is because of absorbance peak of fiber in that range of frequencies. In this range of frequencies, the peaks for PVA, PVA/EFB-2% and PVA/EFB-5% are 1099, 1096 and 1093 cm$^{-1}$ by absorbance of 79.7, 75.57 and 73.34 % respectively. The peak for pure fiber in this range in frequency of 1034 cm$^{-1}$ showing the absorbance of 91.78 % which it affected on composites.

Totally, it could say that there is a small change of absorbance due to fiber content which it becomes clear as the peak of fiber appeared.
4.2. Void content

The presence of void content in the composites significantly reduces the mechanical and physical properties of the composites. During impregnation of fibers into the matrix or during manufacturing of fiber reinforced composites, the trapped air or other volatiles exist in the composites (Jawaid, Khalil, & Bakar, 2011; Khan, Ganster, & Fink, 2009). The most common cause of voids is the incapability of the matrix to displace all the air which is entrained within the woven or chopped fibers as it passes through the matrix impregnation. The void content (%) of composite samples with different fiber amounts are presented in

Figure 4.3. FTIR spectra of PVA and its composites and pure fiber.
Table 4.1. The higher fiber percentage exhibited higher amount of voids (12.68%) compared to two others (8.32% and 1.02%), it’s due to incompatibility between polymer and fibers in higher amounts. Oil palm fibers are porous and the mat is loosely packed hence a large amount of resin would be squeezed out of the mat during pressing molding.

Table 4.2. Void contents of PVA/EFB composite by percentages of 2, 4 and 5.

<table>
<thead>
<tr>
<th>Type of composite</th>
<th>Void content</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVA/EFB- 2%</td>
<td>1.02%</td>
</tr>
<tr>
<td>PVA/EFB- 4%</td>
<td>8.32%</td>
</tr>
<tr>
<td>PVA/EFB- 5%</td>
<td>12.68%</td>
</tr>
</tbody>
</table>

4.3. Hardness and strength

According to (Suwanprateeb, 1998) which preformed polymer hardness test by time dependent, it is observed that the hardness decreased slightly with increasing dwell time. The test performed for two loads in three different dwell times and for each dwell time test performed for 5 times and averages of results are presented in table 4.1.

Table 4.3.0. The hardness of PVA with two load and three different dwell time.

<table>
<thead>
<tr>
<th>Hardness of PVA</th>
<th>load</th>
<th>10(gram)</th>
<th>25(gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dwell time (s)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>6.01</td>
<td>5.98</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>5.83</td>
<td>5.52</td>
</tr>
<tr>
<td>20</td>
<td></td>
<td>5.17</td>
<td>5.31</td>
</tr>
</tbody>
</table>
Table 4.3.1. The hardness of PVA/EFB-2% with two load and three different dwell time.

<table>
<thead>
<tr>
<th>Dwell time (s)</th>
<th>Load</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10(gram)</td>
</tr>
<tr>
<td>5</td>
<td>8.88</td>
</tr>
<tr>
<td>10</td>
<td>8.74</td>
</tr>
<tr>
<td>20</td>
<td>8.38</td>
</tr>
</tbody>
</table>

Table 4.3.2. The hardness of PVA/EFB-5% with two load and three different dwell time.

<table>
<thead>
<tr>
<th>Dwell time(s)</th>
<th>Load</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10(gram)</td>
</tr>
<tr>
<td>5</td>
<td>8.86</td>
</tr>
<tr>
<td>10</td>
<td>8.82</td>
</tr>
<tr>
<td>20</td>
<td>8.80</td>
</tr>
</tbody>
</table>

Polymers are viscoelastic materials, therefore, their behavior is time-dependent. Their properties are not single values, but vary with time under observation. The hardness decreased as the time elapsed. This is analogous to creep behavior when the materials deform under constant load. Additionally, the hardness value produced by the application of the load can be related to the rigidity of the materials. Figure 4.4 showing the indentation of surface which need to major by electronic ruler and calculate the hardness in HV.
After repeating the test for the PVA sample by three load and 3 different dwell time, the result are shown in figure 4.5.0.

Also, for two others sample PVA/EFB-2% and PVA/EFB-5% the test repeated with same condition as PVA and the results are shown in tables 4.5.1 and 4.5.2.
Figure 4.5.1. Effect of load on hardness of PVA/EFB-2% at various dwell times.

Figure 4.5.2. Effect of load on hardness of PVA/EFB-5% at various dwell times.

Furthermore, from the results of HV of samples can use the formula 3.4 which represent the approximate yield strength in MPa. In this case, the average of HV for each samples considered to get the best accuracy that shows in table 4.4.
Table 4.4. Average hardness of three samples in HV and MPa.

<table>
<thead>
<tr>
<th>Sample</th>
<th>load (gr)</th>
<th>Average (HV)</th>
<th>Average (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>PVA</td>
<td>5.67</td>
<td>5.60</td>
<td>5.64</td>
</tr>
<tr>
<td>PVA/EFB-2%</td>
<td>8.56</td>
<td>8.62</td>
<td>8.59</td>
</tr>
<tr>
<td>PVA/EFB-5%</td>
<td>8.82</td>
<td>8.70</td>
<td>8.76</td>
</tr>
</tbody>
</table>

By considering average values of table 4.4 and using equation 3.5, yield strength can be calculated and approximate in table 4.5.

Table 4.5. Yield strength of three samples in MPa.

<table>
<thead>
<tr>
<th>Yield strength</th>
<th>sample</th>
<th>Yield strength (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PVA</td>
<td>22.340</td>
</tr>
<tr>
<td></td>
<td>PVA/EFB-2%</td>
<td>34.051</td>
</tr>
<tr>
<td></td>
<td>PVA/EFB-5%</td>
<td>34.721</td>
</tr>
</tbody>
</table>

As it is shown in table, the PVA/EFB-2% and 5% showing increase about 10 Mpa compare to original PVA sample which it is showing the effect of fibers on PVA.

4.4. Thermal analysis

DSC is one of the common methods used to verify phase separation or polymer–polymer miscibility in any of the blends. Typically, changes in the glass transition temperature of blends versus individual homopolymers are determined. When one of the components is
crystalline, the melting point depression of the polymer may also be used as evidence of the interactions between polymers (Rathna, Jog, & Gaikwad, 2011). Figure 4.4 shows the changes that occurred before and after blending the polymers. Owing to its semicrystallinity, pure PVA recorded a $T_g$ and $T_m$ of 75.7 and 191.6°C, respectively, whereas PVA/EFB-2% formed from PVA recorded unknown $T_g$ and $T_m$ of 193.8°C and for PVA/EFB-5% recorded $T_m$ of 193.34°C without $T_g$. The difference in the melting temperature between pure PVA and composites is about 2 to 4°C. In a nutshell, the fibers only made small change on melting points of composites.

![Figure 4.6. DSC thermographs of pure polymer (PVA) and fiber reinforcement of pure polymer (PVA and PVA/EFB-2&5%).](image)

Thermogravimetry is one of the most widely used techniques to monitor the composition and structural dependence on the thermal degradation of a composite. A typical TGA curve of a
polymer the following weight loss step such as: volatiles (moisture, solvents, monomers), polymer decomposition, change of atmosphere, burning step and residue of fillers or fibers. Figure 4.5 shows the results of thermogravimetric analyses of pure PVA and composites of it. The three curve are almost same and there are not any change in weight loss. The decomposition for all three samples happen between about 290 to 360 °C.

![Figure 4.7.TGA of PVA and composites of it (PVA/EFB-2&5%).](image)

For more detail and to see the weight loss clearly, differentiate of thermogravimetric (DTG) plotted from above data which is shown in figure 4.6. The DTG curve shows an initial and sharp peak between 270 and 370°C with weight loss of up to 60% for all three samples, DTG shows two small peaks in the range of 390 to 540°C which may arise from the loss of chemical bonding water.
Figure 4.8. DTG cure of PVA and composites of it (PVA/EFB-2&5%).

By this mean, for all three samples results are very close and almost same which fibers in PVA not effected for thermal resistant of samples. The pure PVA and PVA/EFB-2&5% gave same result for TGA which looks logical due to fibers are flammable and burn quickly.
CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusion

The research was carried out to investigate palm fibers as reinforcement in biopolymer composite. The study was on preparation of samples and mechanical properties of composite and the effect of amount of fiber content that contribute to the increasing of hardness Vickers (HV) of composite. For material application in industry and etc, chemical resistance test, FTIR and water absorption test was performed for each of samples. The void content of samples was also determined due to effect of it on mechanical properties. Furthermore, for the new composites and to get know more about it thermal test such as DCS and TGA was performed. The samples observed by FESEM to see how fibers are mixed. The objectives of study were achieved.

The hardness test of composites was performed after samples are prepared from special process in specific size to get accurate results. The results showed the increase of hardness of composites by increasing of fiber content. Also, hardness Vickers test was performed in different dwell time to accurate the results due to creep and vicoelastic properties of polymers which show decrease hardness as the time elapsed.

Chemical resistance test was observed that matrix showed better chemical resistant to all chemicals except resistance to HCL which all the samples was solvate in it during the 24 hours.

Hydrophilicity of fibers caused the higher water absorption in composite compare to original polymer. It means that the composite with high content of fiber had higher water absorbance.
The presence of void content in the composites is important due to its effect on mechanical properties of it. The most common cause of voids is the incapability of the matrix to displace all the air which is entrained within the woven or chopped fibers as it passes through the matrix impregnation. The void content (%) of composite samples increases with amount of fibers which means higher fiber content has higher void content.

The last parts of experiments presented the thermal properties of composite which used TGA and DSC to see the changes between pure PVA and composite of it. Totally, the effect of fibers in composites for thermal resistance and thermal properties of it were not considerable. The changes of melting point about 2 to 4°C was observed during the test this because of flammability of fibers and less heat-resistant which TGA test proved the accuracy of the theory.

In conclusion, natural fibers are increasing mechanical properties of polymers and are the cheapest and easiest way to improve the property of polymers. Also, natural fibers are degradable so they do not damage or pollute environment as they recycle. The noticeable point for natural fibers is they are inflammable so the composites which made by them cannot be used in places which need heat-resistant.

5.2. Recommendation

The following recommendation can be suggested from this study:

1. Apply the suitable method on designing the mould and manufacturing samples during press.

2. Add some others kinds of natural fillers such as talc or calcium carbonate to composites for improving the properties especially heat-resistant of it.
3. Apply suitable treatment of natural fibers for improving strength of fibers. In this case, improve fiber matrix interfacial bonding of composites, novel processing techniques, chemical and physical modification methods are developed. The treatment can enhance interfacial adhesion between fiber and polymer matrix which could be improved the mechanical properties such as hardness or yield strength.


